AN EVALUATION OF THE USE OF HIGH TEMPERATURE PROCEDURES

FOR APPLYING DIRECT DYES TO COTTON YARN

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A THESIS

Presented to

the Faculty of the Graduate Division

Georgia Institute of Technology

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Textiles

by

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December 1953

AN EVALUATION OF THE USE OF HIGH TEMPERATURE PROCEDURES

FOR APPLYING DIRECT DYES TO COTTON YARN

APPROVED: ______________________

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Date Approved by Chairman December 18, 1953

ACKNOWLEDGEMENTS

On the completion of this work I wish to express my gratitude to Dr. James L. Taylor of the Georgia Institute of Technology for his most valuable assistance and guidance. I also wish to thank Mr. Alton R. Colcord for his helpful criticism and suggestions.

The material assistance furnished by the Morton Machine Works, Columbus, Georgia; the Swift Manufacturing Company, Inc., Columbus, Georgia; and the personnel of the Atlanta laboratory of E. I. du Pont de Nemours and Company, Inc. is gratefully acknowledged.

To the T. E. Stribling Foundation, I express my sincere gratitude for the graduate fellowship which made this work possible.

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AN EVALUATION OF THE USE OF HIGH TEMPERATURE PROCEDURES FOR APPLYING DIRECT DYES TO COTTON YARN

SUMMARY

In this problem an attempt is made to evaluate the use of the high temperature-pressure method in the application of direct dyes to cotton yarns to determine whether or not any advantage will accrue in increased resistance to fading by light and washing, reduction in the time required for dyeing and reduction in the amount of dyestuff to be used.

A laboratory model of a one-pound package dyeing machine was modified in order that dyeings could be made at the boil and at elevated temperatures under comparable conditions. Comparative dyeings with direct dyes were made at 212° F. and at 250° F. The dyes were selected on the basis of their rated fastness to light and washing. The following dyestuffs were used:

Pontamine Blue BB Super Concentrated

Pontamine Fast Rubine B Concentrated

Pontamine Fast Orange EGL.

An additional series of high temperature dyeings utilizing Pontamine Fast Orange EGL was made in which variations in time of dyeing and concentration of dyestuff were introduced. In this series of dyeings, a cooling cycle was incorporated in the dyeing procedure in an effort to effect better exhaustion of the dye bath.

The results of this investigation indicate that dyeing at

elevated temperatures has no deleterious effect on the strength of the yarn, and the observed loss in strength apparently was caused by chemical action of the dyestuffs employed.

No increase in resistance to fading by light and washing was noted in the yarn dyed at elevated temperatures. Studies of the dyed yarns failed to indicate that additional penetration or exhaustion is obtained by dyeing with direct dyes at elevated temperatures. The conclusion is reached that any increase or decrease in these factors must be attributed to the chemical composition of the individual dyestuffs.

The results obtained in dyeing yarn at 250° F. under variable conditions of time and concentration in a cooling dye bath indicate that a reduction of 20.0 per cent in time of dyeing and a decrease of 12.5 per cent in the amount of dyestuff required may be effected by the use of high temperature procedures.

CHAPTER I

INTRODUCTION

The craft and art of dyeing has been practiced in one form or another for thousands of years. In fact, it is one of the oldest achievements of mankind. The earliest attempts at dyeing made use of various berries, herbs and barks to stain the materials to be colored. These methods naturally restricted the range of colors to a very few.

The discovery by Perkin of the synthetic dye "mauve" in 1856 set off a feverish investigation by the world's chemists to produce a whole range of synthetic colors. This thirst for new dyes has continued to the present day when we find thousands of dyestuffs sufficient to produce any desired shade or tint on any fibrous material.

The earlier dyestuffs were of the class commonly known as "direct dyes" and were widely used, particularly on cotton goods. However, although these direct dyes were, and still are, extremely simple of application, they are for the most part relatively sensitive to light and washing.

Within the past decade a large number of synthetic fibers have been introduced, in addition to rayon and nylon. Some of these newer synthetics have posed considerable problems to the dyer. In the case of several synthetic fibers, the use of high temperatures and pressure have been required to obtain satisfactory dyeings of adequate fastness properties and at a reasonable cost. The high temperature-pressure apparatus proved to be so satisfactory that textile manufacturers have become widely interested in applying the method of dyeing to all classes of fibers, utilizing practically all classes of dyes.

The problem. In this problem, an attempt is made to evaluate the use of the high temperature-pressure method in the application of direct dyes to cotton yarns to determine whether any advantage will accrue in increased resistance to fading by light and washing, reduction in the time required for dyeing, and reduction in the amount of dyestuff to be used.

Direct dyes are utilized on cotton yarns and fabrics quite extensively, particularly where lightfastness or washfastness is not necessarily required. The usual method of applying direct dyes at the boil is a time consuming operation, and a large portion of the dyestuff is wasted since the rates of exhaustion are none too good. Since dyestuffs and labor are both rather expensive, a reduction of either of these costs is of prime interest to the manufacturer.

Review of the literature. Practically every textile organization of size in this country is today conducting research and experimentation on dyes and methods of dye application. A large amount of this research is devoted to high temperature dyeing under pressure. However, very few reports of the results obtained have been published.

The Du Pont Company has recommended and endorsed the use of high temperature equipment on a production scale, particularly for their newer synthetic fibers. In addition, the development of the Barotor has provided a method for dyeing piece-goods in open width at high temperatures under pressure.(1)

The use and construction of a new high temperature, continuous dyeing machine developed by the Bachmann Uxbridge Worsted Corporation is described by Zimmerman.(2) This equipment consists of two main parts, a padder and a pressure chamber for dyeing and developing. After being padded, the cloth passes into the pressure dyeing chamber through a set of sealing rolls which were designed to hold back the high pressures while in motion.

Drijvers(3) made a study of high temperature dyeing on the major classes of fibers and stated that a new type of machine was required to permit the practical use of this method at the industrial level. He described the construction of a machine designed by Baudouin Steverlynck of the Teinturerie de Groeninghe a Courtrai in Belgium. Essentially this new machine featured an enclosed expansion chamber, together with provisions for a pressurized addition chamber and a sampling chamber. It was stated that the air trapped in the enclosed expansion chamber exerted sufficient pressure to overcome the decrease in pump pressure because of back pressure encountered upon raising the temperature of the dye solution above the boil.

Virtually the same recommendations are made in a staff article appearing in the September, 1953 issue of Textil Praxis. A machine of the same type is described as having been manufactured by the H. K. Krantz Maschinenfabrik of Aachen, Germany. The advantages denoted for this machine are substantially the same as those proposed by Drijvers(4).

An improved model of the machine designed by Steverlynck has now been placed in commercial production by a manufacturer in Courtrai, Belgium. Marketed as the "Static-Process Steverlynck", this system controls static pressure from an outside source independent of internal temperatures; has a sampling kier that can be used at any time without stopping the complete unit; eliminates the difficulties previously caused by high local temperatures near the stock or yarns by the installation of a heat-exchanger unit between the circulating pump and the dye kier; and has a special low-pressure circulating pump that prevents much of the channeling and felting caused by high-pressure pumps.(5)

Royer(6) and his associates made a series of studies of the use of high temperatures in dyeing a number of different natural and synthetic fibers. A small laboratory non-continuous dyeing unit was designed and built for dyeing at temperatures up to 300° F. under air pressure of fifty pounds per square inch. The apparatus consists of two chambers, and in operation the solution or developing bath is placed in the lower chamber. A platform-valve, which separates the two chambers, is pulled into place and held there by applying fifty pounds air pressure to the lower chamber. The dye solution is then heated by electrical coils which surround the lower chamber. The sample to be dyed is mounted on a small holder and then placed on the platform-valve which separates the two chambers. When the dye bath has reached the desired temperature, the cover is fastened on the top chamber, and compressed air is let into the top chamber until the pressure in the upper chamber equals that in the lower. At this point, the platform-valve drops, permitting the sample to fall into the dye bath. The sample is agitated during dyeing by rocking the unit manually.

This apparatus is of great value for analytical work since it permits entering the samples into the dyeing bath at the desired dyeing temperature without pre-heating or steaming. In addition, provision is made for quick removal of the sample from the dye bath and rapid cooling.

From their studies, Royer and his associates found that in dyeing cellulosic fibers such as cotton and wool, the increased temperature caused more rapid diffusion and leveling of dyes but a lesser exhaustion of the dye bath. The test dyeings were performed at 270° F. for fifty seconds under air pressure of fifty pounds per square inch. In general it was found that because of the short times of contact at the higher temperatures, the same extent of dyeing could be obtained without greater damage than is obtained for the same reaction during a longer dyeing time at lower temperatures. It was indicated that possible changes in tensile characteristics could be attributed to the reaction of the dye with the fiber, and further that the same damage would occur regardless of the temperature at which the reaction took place.(7)

Drijvers(8) has shown that when using the high temperature dyeing process, each dyestuff used had a characteristic temperature at which complete equilibrium was reached, and increasing the temperature beyond that critical temperature caused the equilibrium curve to decrease. From a series of studies made on this dyeing phenomenon, he has recommended that the temperature be raised to the critical temperature as rapidly as possible to obtain the desired equilibrium, and then introduce a cooling cycle to obtain maximum exhaustion of the dye bath. In this connection, Drijvers has postulated that a major portion of the dye goes on the fibers during cooling of the bath.

CHAPTER II

THEORETICAL CONSIDERATIONS

Any analytical examination into methods of dyeing and the properties of dyed textile materials requires that consideration be given to the molecular construction of the fiber and the theories of dyeing.

Structure of cotton. The natural cotton fiber has a lustrous, creamy white color and appears as an elongated hollow tube with a narrow canal or lumen down the center. Its longitudinal appearance is that of a flat tape or ribbon with regular convolutions throughout its length. In cross-section, the immature fiber appears as a thin-walled collapsed ring, the thickness of the fiber wall increasing and the size of the lumen correspondingly decreasing with maturity.(9)

Chemically, the natural cotton fiber is composed of about 97 to 99 per cent cellulose, the remaining portions consisting of pectins, waxes and soluble impurities.(10) A great deal of research work has been done in the last two decades in an effort to determine the exact structure of the cellulosic portion of cotton. It has become generally accepted that cotton is built up from very long thread-like molecules, the basic component of which is cellobiose.(11)

When textile fibers are examined by X-rays, they are found to produce regular diffraction patterns similar to those produced by the rotation of crystals. The patterns are usually accentuated by stretching the fibers, since this increases the orientation of the molecules.

According to Vickerstaff(12), the formation of these patterns proves that in certain parts of the fiber the molecules are arranged in an orderly and regular manner. These oriented portions, which have been called "crystallites" or "micelles", cannot comprise the whole of the fiber, since the physical properties of fibers differ widely from those which would be exhibited by crystals. Consequently it is necessary to suppose that the micelles are embedded in material of a different character. At one time it was suggested that the crystallites were discrete and separate and were like bricks embedded in a matrix of different material. Now, however, the most generally accepted view is that the crystallites are not discrete but merge gradually into a more disordered or amorphous portion of the fiber which is chemically identical with the crystallites. It is further believed that one single-chain molecule may pass through and be part of two or more crystallites.

The fiber thus consists of dense, tightly packed and oriented regions interspersed with less-dense amorphous regions, and the latter may be regarded as forming a system of interlocking pores or capillaries throughout the mass. The relative amount of crystalline and amorphous material has been the subject of much study but no very definite conclusions have been reached.

<u>Theory of dyeing</u>. The behavior of native cellulose to dyestuff solutions has been the subject of numerous investigations. Similarly, numerous theories of dyeing have been suggested, although most of the older theories have now been abandoned. The elucidation of the fine structure of the textile fibers has influenced and furthered this development to a great extent. Today, it is generally believed that the cotton dyes are absorbed from their aqueous solutions, and that the dye

forms a molecular compound with the cellulose. Heuser(13) proposes that the forces which are operative in binding the dye to the fiber are probably the same that are instrumental in holding the individual chain molecules together. These forces may be operative as secondary valences or as co-ordinative hydrogen bonds between the hydroxyl groups of the cellulose and certain polar groups of the dye molecule.

The capillary system of the fiber on the one side and the particle size of the dye molecules on the other are of great importance because the dye molecules must penetrate the capillaries to reach the interior of the fiber, and only dyes of sufficiently small particle size can enter the finer capillaries. Even in the swollen state, which is a prerequisite for dye absorption, the chains within the crystalline regions of the micellar system are probably packed too closely to be reached by the common dye molecules; for this reason penetration is generally limited to the amorphous regions.

Drijvers(14) also has pointed out this capillary action by stating that the dye is absorbed not only on the surface but is distributed through the whole fiber. The rate of absorption, depending upon such factors as the ratio of dye bath to fiber, the temperature and the salt concentration of the dye bath, decreases until an equilibrium is reached. In this state, the amount of dye absorbed by the fiber is in equilibrium with the concentration of the dye dissolved in the bath.

Heuser(15) has stated that the reactivity of mercerized and the various types of regenerated cellulose is greater than that of the untreated, original material. Mercerization is essentially a controlled swelling of cellulose, and the results of such swelling can be seen in the more uniform appearance of the cotton fiber in cross-section and in the deconvoluted appearance of the fiber. The increased swelling caused by mercerization, and the consequent increase in size of the capillaries, probably accounts for the greater absorption of dyestuffs by mercerized cotton.

Drijvers(15) explains current dyeing theories by stating that dyeing takes place in three distinct phases: the diffusion of the dye in the bath towards the surface of the fiber; the adsorption of the dye on the surface of the fiber; and diffusion of the dye into the fiber. In machine dyeing with the dye liquor being circulated through the package of yarn, the outer layers of yarn reach a dye equilibrium with the dye bath before the inner portions of the package. Because of this fact, the unequal equilibrium causes the dye on the outer portion of the package to go back into solution again. As the dye equilibrium is shifted, the inner portions of the package again begin to absorb dyestuff until the absorption of dye is uniform throughout the package of yarn, at which time complete dyeing equilibrium will have been attained. These facts may be incorporated in the following fundamental principle, as proposed by Drijvers:

> "In a dynamic system (dye solution/mass to be dyed) no change whatever, so far as the practical exhaustion of the bath is concerned, can be achieved as long as tinctorial equilibrium has not been reached between the entire mass and the whole of the bath."

Although the dye equilibrium can be shifted by the presence of electrolytes, the rate at which they are reached is principally a function of the temperature.

In a logical application of the foregoing hypotheses, it can be concluded that an increase in swelling of the cotton fiber could be obtained through the use of higher temperatures than those normally used, and further that increased dye absorption could result from the enlargement of the inter-micellar capillaries or the possible increase in the overall proportion of amorphous material caused by such swelling.

CHAPTER III

INSTRUMENTATION AND EQUIPMENT

The following is a complete list of the machines and instruments utilized in conducting this investigation:

Modified Morton One-pound Package Dyeing Machine

Lumetron Colorimeter, Model 402-E

Atlas Fadeometer

Atlas Launderometer

Fidelity Ribber, 176 Needles

Suter Single End Break Machine

Goodbrand Twist Counter

Foster Model 102 Winder

Christian-Becker Chainomatic Analytical Balance

Brown and Sharpe Yarn Reel.

CHAPTER IV

EXPERIMENTAL PROCEDURE

PRELIMINARY TESTS AND PREPARATION FOR DYEING

As a preliminary to dyeing, information was obtained regarding the yarn number or count, twists per inch, and breaking strength of the yarn. These tests were conducted under standard conditions (65 per cent relative humidity and 70° F. temperature). The yarns were stored in the laboratory for a minimum of twenty-four hours in order that standard conditioning would be attained.

Yarn number or count. The yarn number or count of the yarn was determined by reeling 120-yard samples and weighing the samples on the Christian-Becker Chainomatic Balance. The length and weights were put in the following formula to determine the counts:

$$\text{Yarn number} = \frac{\text{Length in yards}}{\text{Weight in grains}} \times \frac{7000}{840}$$

Two samples were taken from each of ten cones of the yarn, each cone containing one and one-half pounds of yarn. The average yarn number obtained for the twenty samples was taken as the count of the yarn used in this experiment. The results are shown in Table 1.

Twists per inch. The untwist-twist method was used to determine the twist per inch of the yarn. A ten-inch sample was inserted in the jaws of the Goodbrand Twist Counter. The tension on the yarn was applied so that the indicator was at the top position. As the yarn was untwisted, the indicator dropped to the bottom position. As the untwisting continued, the point was reached such that all the twist had been removed; and continued turning of the jaws at one end tended to insert twist in the yarn again to the extent that the yarn contracted and raised the indicator to its original position.

Since the reading on the scale indicates the total turns required to remove all the twist and re-insert this same amount of twist in a ten-inch sample, it is divided by two and the resulting figure divided by ten to obtain the number of turns per inch. Two samples were taken from each of ten cones of yarn, each cone containing one and one-half pounds of yarn. The average of the twenty twist tests was taken as the twist per inch of the yarn. The results are shown in Table 2.

Breaking strength. Information regarding the strength of the yarn was obtained to form the basis for determination of any possible damage to the yarn as a result of dyeing. Yarn strength was tested on the Suter Vertical Single Strand Yarn Tester (oil plunger type). The tests were made with the jaws set ten inches apart, and the rate of descent of the lower jaw was set at twelve inches per minute. Care was taken to insure that no twist was lost in inserting the specimen in the jaws of the tester; and if the yarn broke at the jaw, the results were discarded. Four samples were taken from each of ten cones of yarn, and the average of the forty breaking strength tests was taken as the breaking strength of the yarn. These results are shown in Table 3.

Winding the yarn. The package dyeing machine used in this experiment was designed to accommodate one-pound packages of yarn. It was therefore necessary that the yarn be re-wound on metal dyeing tubes, each tube to contain one pound net of yarn. Winding was performed on the Foster Model 102 Winder. Care was taken to insure that as soft a package as possible was prepared. This necessitated as little tension as possible on the yarn during winding, and an 88-gram weight was used to apply this small amount of tension on the yarn.

INSTRUMENTATION AND EQUIPMENT

Modified Morton one-pound package dyeing machine. The machine selected for use in dyeing the yarn in this problem was the Morton one-pound package dyeing machine. This particular model is the standard machine designed for sample dyeings in the laboratory. It consists of a dye kier with a closed cover into which a one-pound package of yarn is placed. The yarn is wound on a special stainless steel tube which has regularly spaced holes punched in it. The tube fits over a spindle which also has regularly spaced holes. The dye kier is connected with suitable piping to an open expansion tank which allows the level of the dye liquor to rise in the expansion tank as the dye liquor is heated. A centrifugal pump in the pipeline between the expansion tank and the dye kier forces the dye liquor through the one-pound package of yarn with a pressure of twenty pounds. Provision is made for periodically reversing the flow of dye liquor from outside-in to inside-out.

The source of heat for the machine is a steam jacket around the dye kier. The temperature of dyeing is controlled manually, and the temperature is measured by a thermometer mounted in the cover of the dye kier.

In a preliminary investigation of this problem, a number of sample dyeings were made to check the suitability of the machine. Ordinarily, the machine is sued for dyeing at the boil, in which case air locks in the piping can be eliminated by means of the bleeder valve placed in the top of the dye kier. However, for high temperature dyeing under pressure, the machine must be completely closed off, and it is impossible to bleed air or accumulated steam out of the machine without also permitting an unmeasurable portion of the dye liquor also to escape.

It was also determined that if the machine were to be utilized in its normal form, it would be necessary to perform all the dyeings, both high temperature and at the boil, under closed conditions. This would be necessary since the dye solution can only be added by means of the expansion tank. When dyeing at high temperature under pressure, the dye is dissolved in a given quantity of water and the machine is closed off when the proper temperature is reached. Inasmuch as the valves for pressurizing the machine are below the expansion tank, approximately one-third of the dye liquor is left in the expansion tank and not utilized for dyeing. In order to make comparable dyeings, it was thus necessary that the dyeings at the boil be made under closed conditions also. Such a procedure would be extremely difficult to execute because the method of heat control is very slow to react, and it would be almost impossible to maintain the temperature of the dye liquor at or just below the boil.

In dyeing under pressure with the machine in its original form, when air locks formed in the pipeline, the pump pressure became greatly reduced and even completely nullified in some cases. When dyeing at high temperature, the dyeing proceeded adequately for a few minutes, but the pump pressure gradually dropped until only the pressure of the dye liquor remained. This was observed by stopping the motor to the pump and reading the pressure of the liquor on the gauge. Ordinarily this gauge indicated, at 250° F., a pump pressure of twenty pounds and a dye liquor pressure of twenty to twenty-five pounds.

Because of the above objectionable features, it was determined that it would be necessary to make certain revisions in the machine in order that more comparable results could be obtained and in order that the number of possible variables would be reduced. It was decided to make the following changes:

1. Pressurize the expansion tank by fitting it with a cover.
2. Install a heating coil in the expansion tank.
3. Install a thermometer in the pipeline.

These changes were designed to achieve the following results:

1. Permit comparable dyeings to be made at the boil with the expansion tank open and at high temperature under pressure.
2. Utilize all the dye liquor in the machine instead of isolating an unused portion in the expansion tank.
3. Maintain constant pump pressure at all times by utilizing the air trapped in the upper portion of the closed expansion tank to exert a counter-pressure against the expansion of the dye liquor and force any air trapped in the system through the machine until it reached the air space where it could be absorbed. Any steam formed by the high temperature would also be liberated into the air space instead of being forced back into the dye liquor to prevent the pump's operating at constant pressure.
4. Permit more rapid heating and better heat control,

particularly for high temperature dyeing under pressure.

5. Permit more reliable temperature readings to be obtained.

After making the above changes, test dyeings revealed that all of the prior objections to the machine had been overcome. Photographs of the revised machine, showing the details of the covered expansion tank and the new thermometer location, are shown in Figures 1 and 2.

The Lumetron Colorimeter. The Lumetron Photoelectric Colorimeter, Model 402-E, is a device for measuring the amount of light transmitted by a sample solution as compared with a standard solution. Essentially, it consists of a white light source which, when projected through a suitable filter, is converted into monochromatic light. The monochromatic light passes through the sample being tested, and the light transmitted by the sample strikes a photocell which converts the light transmission into an electrical impulse which is passed on to a galvanometer. By means of an adjustable dial, the per cent light transmission of the sample can be read directly. This machine is illustrated in Figure 3.

The per cent light transmission obtained through the use of the colorimeter can be easily converted into dyestuff concentration by applying the Lambert-Beer Law.(16) In order to apply this law, standard solutions of the dyes to be utilized were prepared and tested on the colorimeter. A straight line was obtained when the per cent light transmissions were plotted against concentrations on semi-logarithmic coordinate paper. Thus the per cent light transmissions obtained for the dye baths before and after dyeing could be read directly by plotting the readings on the straight line curves of the standard solutions of known concentrations. Figures 3, 4 and 5 illustrate the plotted curves for standard solutions of the dyes used in this experiment.

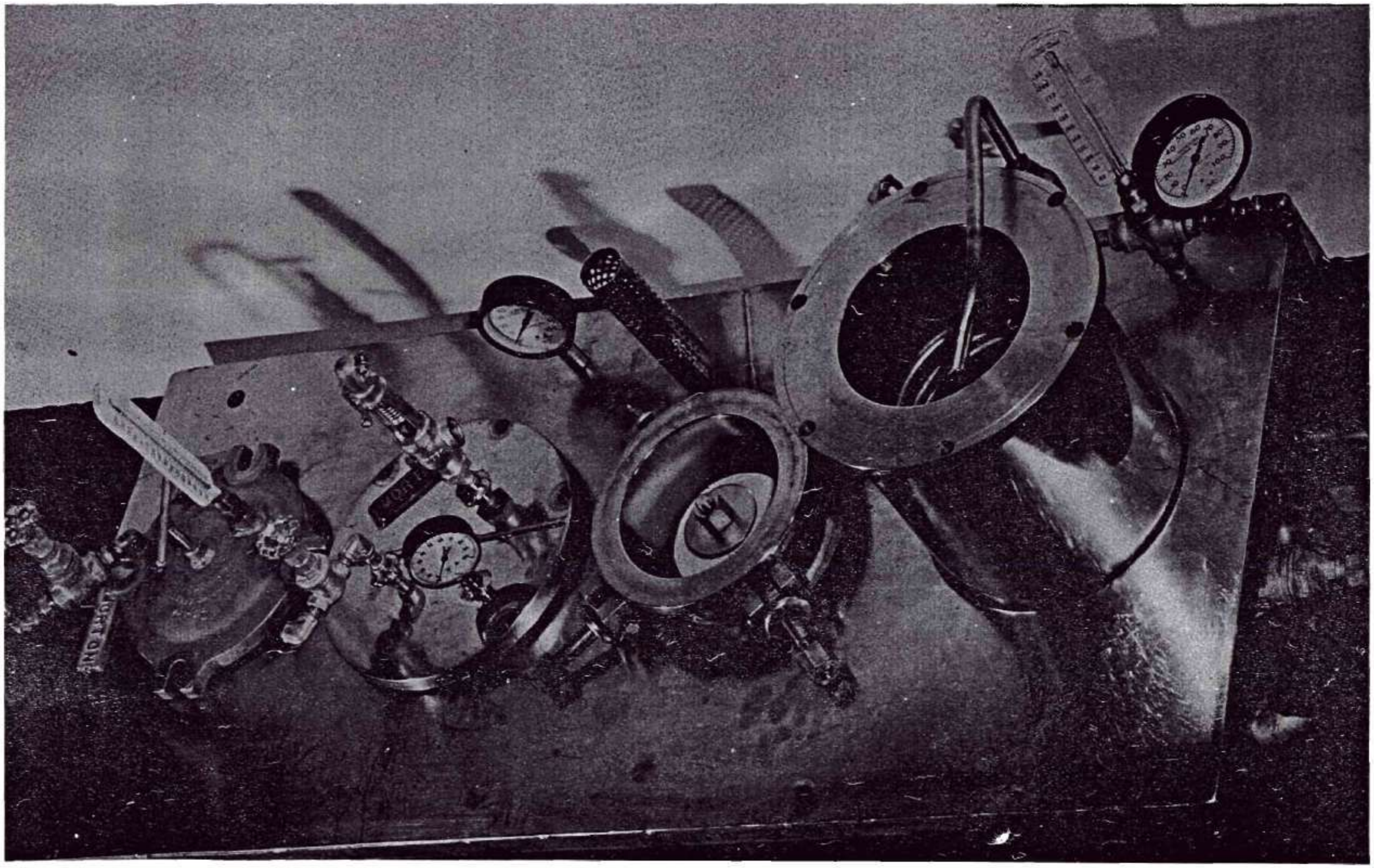

Figure 1. Modified Morton One-Pound Package Dyeing Machine (Top View)

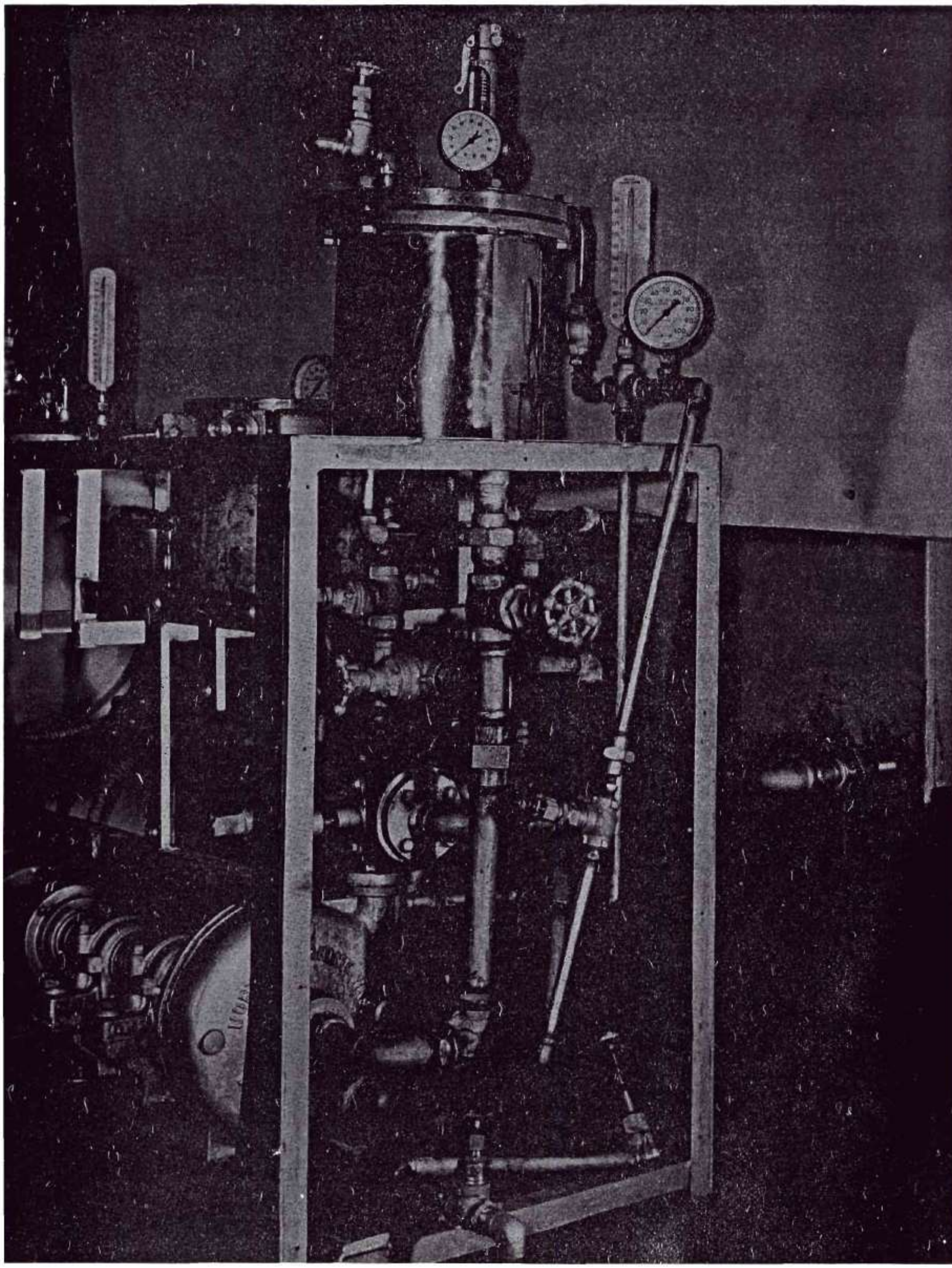

Figure 2. Modified Morton One-Pound Package Dyeing Machine (End View)

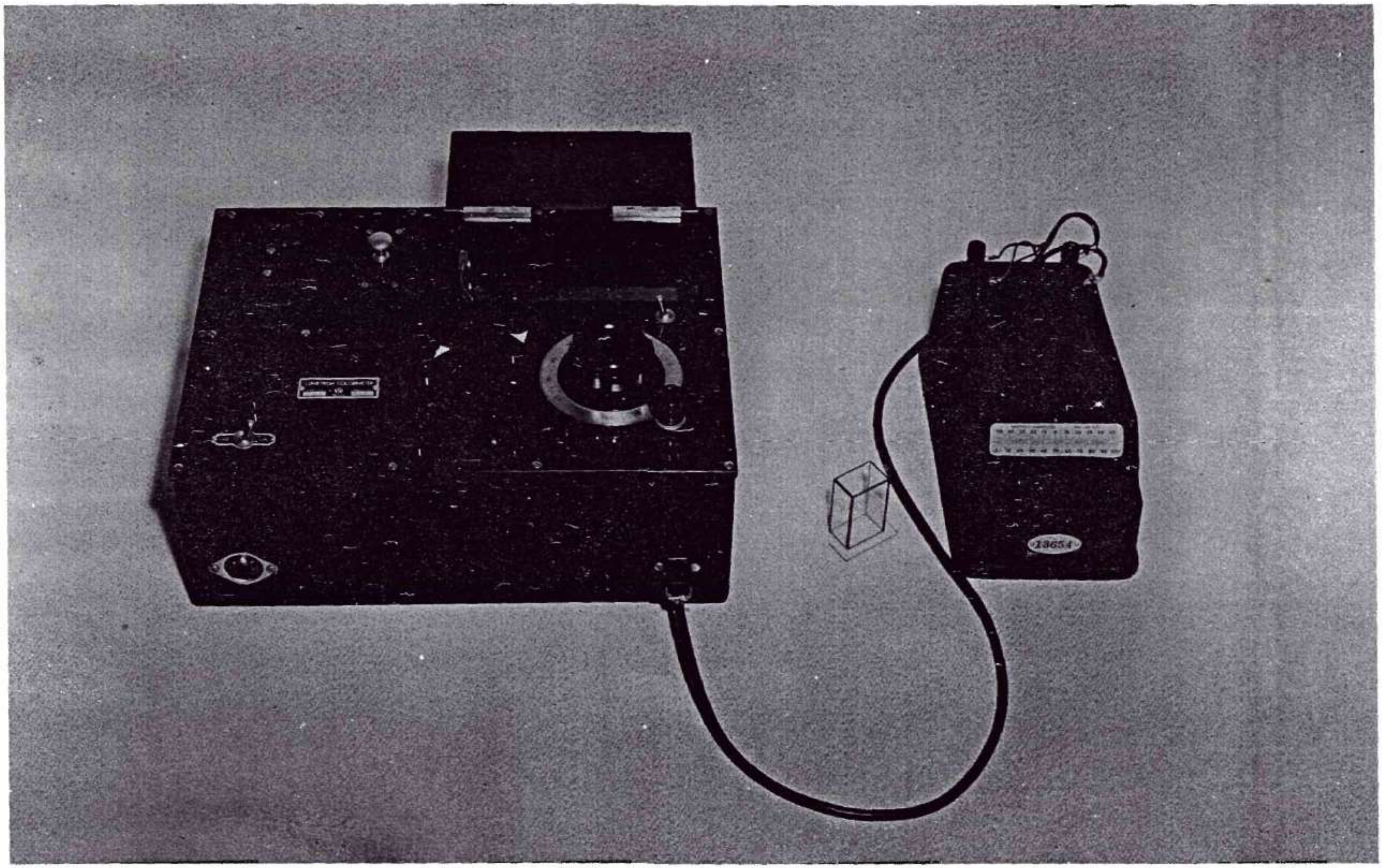

Figure 3. Lumetron Colorimeter, Model 402-E.

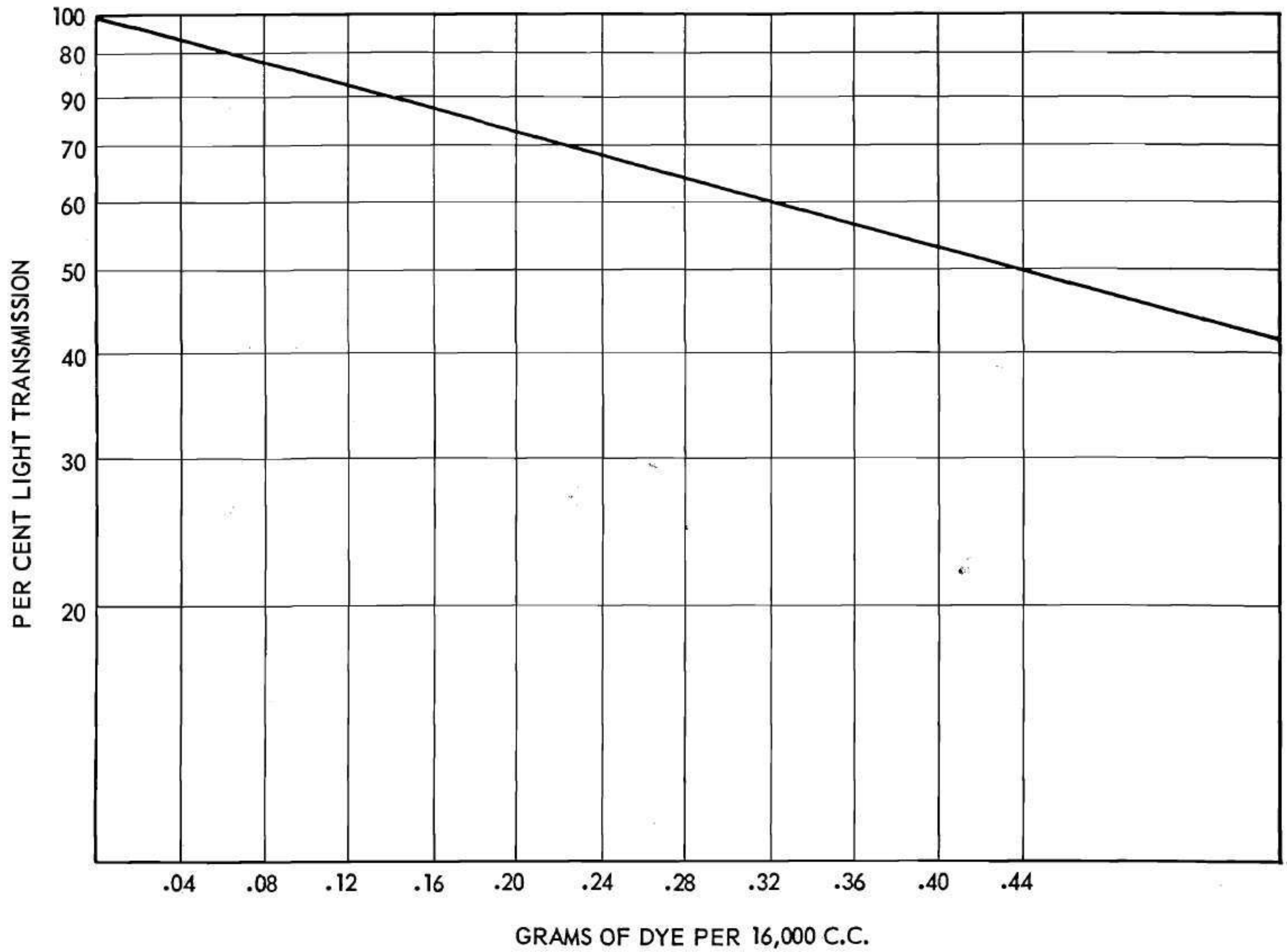

Figure 4. Concentrations of Pontamine Blue BB Super Concentrated for Various Light Transmission Percentages.

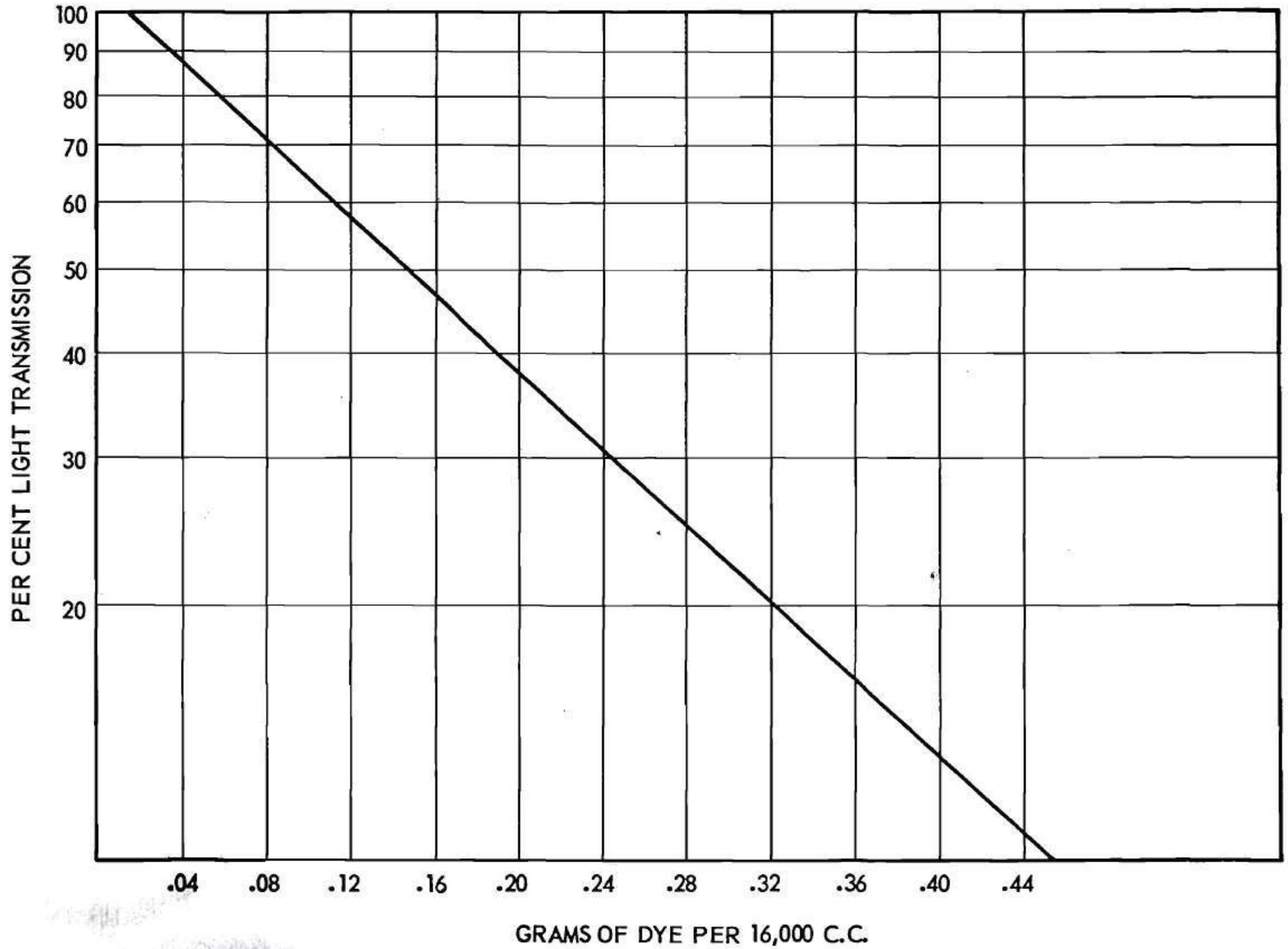

Figure 5. Concentrations of Pontamine Fast Rubine B Concentrated for Various Light Transmission Percentages.

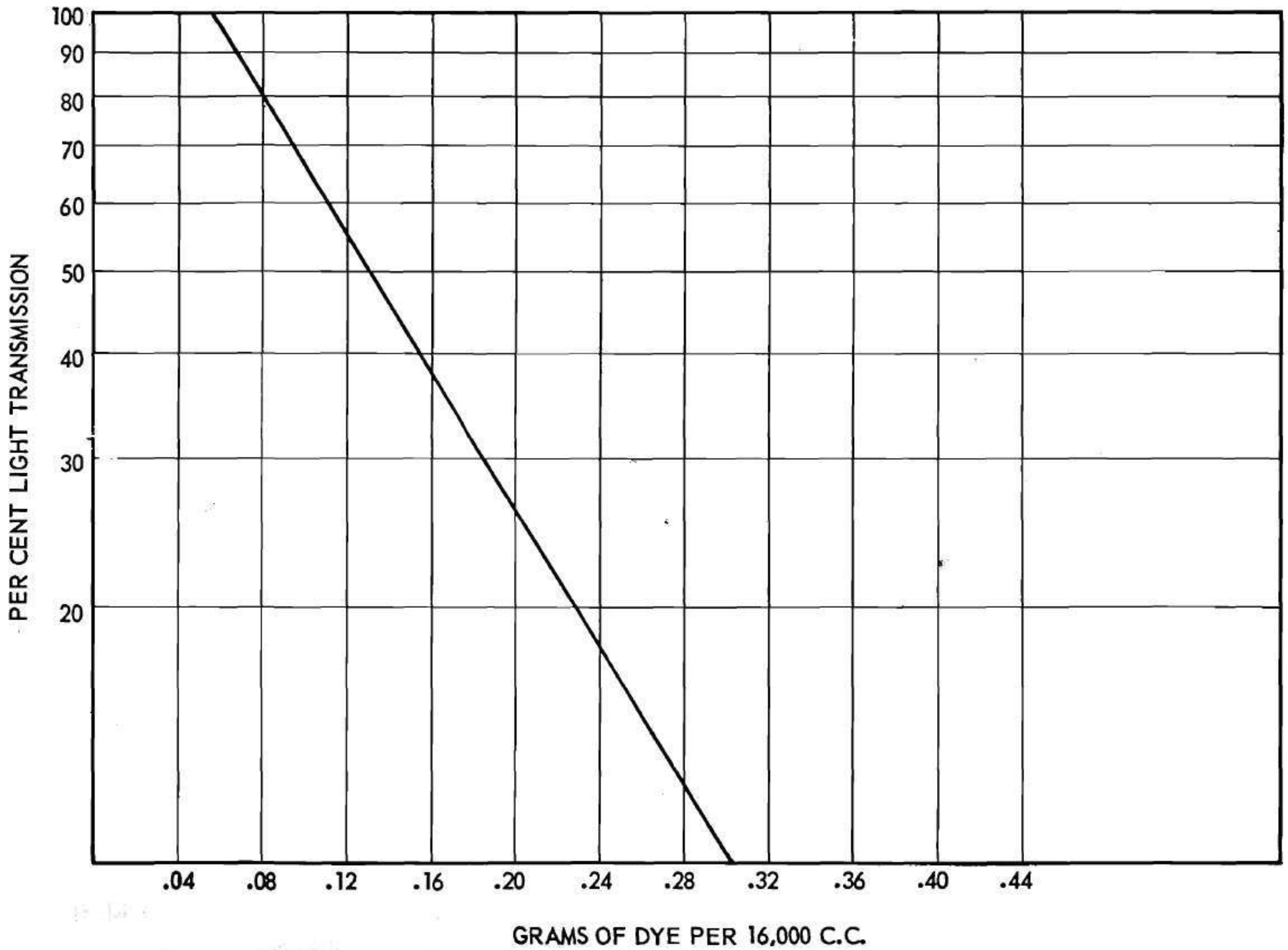

Figure 6. Concentrations of Pontamine Fast Orange EGL for Various Light Transmission Percentages.

DYEING THE YARNS

Types of dyes used. The dyes used in this experiment, together with the fastness characteristics specified by the dye manufacturer, were as follows:

Pontamine Blue BB Super Concentrated (C.I. 406)

Lightfastness (3 per cent dyeing): 1 (1.25 to 2.50 Fadeometer hours)

Washfastness (A.A.T.C.C. Test No. 2): 1W (Poor)

Pontamine Fast Rubine B Concentrated (Pr. 539)

Lightfastness (3 per cent dyeing): 4 (10 to 20 Fadeometer hours)

Washfastness (A.A.T.C.C. Test No. 2): 4W (Good)

Pontamine Fast Orange EGL (Pr. 72)

Lightfastness (3 per cent dyeing): 5 (20 to 40 Fadeometer hours)

Washfastness (A.A.T.C.C. Test No. 2): 4W (Good)

Pontamine Blue BB Super Concentrated has the chemical formula $C_{32}H_{20}N_6O_{14}S_4Na_4$. An attempt was made to obtain the chemical formulas of the other two dyes but no information was available except the fact that they are of the stilbene type.

The concentration of the dye solution in this series of experiments was two per cent based on the weight of the goods.

Dyeing at the boil. Dyeing at the boil was carried out in the usual manner with the open expansion chamber. The yarn was first wet out for twenty minutes at 180° F. with a solution of 1.0 per cent Merpol F and 1.0 per cent trisodium phosphate in a liquor ratio of 16 to 1. The direction of flow was reversed every two to three minutes. The bath was then dropped, and the yarn was rinsed with cold running water until the solution became clear.

The drain was then closed and the dye bath was set with one-half of the dye concentration, running cold outside-in. At the end of five minutes, the remaining half of the dye concentration was added, still running cold.

The temperature of the bath was raised to 180° F. in ten minutes, at which time fifteen per cent salt was added. Circulation was continued at 180° F. for five minutes, and then the temperature was raised to 212° F.

Dyeing was continued at the boil for a period of twenty minutes, after which the bath was dropped and the yarn rinsed with cold running water until the bath was clear. The excess water was then removed in the extractor and the yarn dried in the circulating oven.

Dyeing at high temperature under pressure. The procedure for high temperature dyeing was practically the same as that utilized for dyeing at the boil. The yarn was wet out in the same manner with Merpol F and trisodium phosphate. The dye bath was set with half the dye concentration and circulated outside-in cold for five minutes. Then the remaining half of the dye concentration was added, and the temperature of the dye bath was raised to 180° F. in ten minutes.

At this point, fifteen per cent salt was added and open circulation continued for five minutes at 180° F. Then the cover for the expansion tank was fastened securely in place, and the temperature of the dye bath was raised to 250° F. in ten minutes. It was necessary to utilize the heating coil in the expansion tank to attain this higher temperature in the short time. Dyeing was continued at 250° F. for a period of twenty minutes, after which time the dye bath was dropped.

The yarn was then rinsed, extracted and dried in the same manner as that utilized in dyeing at the boil.

Results of dyeing. The resultant dyeings of both methods produced good dyeings of uniform shades throughout the one-pound packages. The high temperature dyeings using Pontamine Fast Rubine B Concentrated were of a slightly heavier shade than those produced at the boil using the same dye. On the other hand, the use of Pontamine Fast Orange EGL and Pontamine Blue BB Super Concentrated produced heavier shades at the boil than with the use of high temperature procedures.

The exhaustion percentages for the various dyeings, as determined by colorimetric methods, are shown in Tables 4, 5 and 6 of the appendix.

TESTS ON DYED YARN

Backwinding. Prior to testing the dyed yarn, it was necessary to re-wind the yarn onto cones since the one-pound packages were not suitable for yarn testing. It was desired to evaluate the deleterious effect of the high temperature dye solution on the outer, middle and inner portions of the one-pound package. For this reason, each one-pound package was divided into three portions of equal thickness on the package, and each portion was re-wound onto a separate cone. Thus three samples from each one-pound package were made available for testing purposes.

Breaking strength tests. The average breaking strength of the dyed yarn was determined by making eight single strand breaks from each of five cones of dyed yarn, a total of forty breaks for each evaluation. The average of the forty breaks was taken as the breaking strength of the dyed yarn. The results of these breaking strength tests are shown in Tables 7 through 24 of the appendix.

<u>Launderometer tests</u>. Cloth knitted from the test yarn was cut into 2" x 4" samples, and a 2" x 2" square of bleached sheeting was sewed to the face of each sample. These samples were then placed in the Launderometer and subjected to A.A.T.C.C. Test No. 2.(17)

<u>Fadeometer tests</u>. Cloth knitted from the test yarn was cut into $2\frac{1}{2}$" x 6" samples which were placed in test masks and subjected to exposure in the fadeometer.(18) The times of exposure, selected to cover the lightfastness ratings specified by the dyestuff manufacturer, were as follows:

<u>Pontamine Blue BB Super Concentrated</u>

Exposure No. 1.	1.25 hours
Exposure No. 2.	2.50 hours
Exposure No. 3.	3.75 hours
Exposure No. 4.	5.00 hours

<u>Pontamine Fast Rubine B Concentrated</u>

Exposure No. 1.	5.00 hours
Exposure No. 2.	10.00 hours
Exposure No. 3.	15.00 hours
Exposure No. 4.	20.00 hours

<u>Pontamine Fast Orange EGL</u>

Exposure No. 1.	20.00 hours
Exposure No. 2.	30.00 hours
Exposure No. 3.	40.00 hours
Exposure No. 4.	50.00 hours

<u>Microscopic examination</u>. Fibers from the dyed yarns were examined under a microscope both longitudinally and in cross-section in an effort to determine whether or not better penetration and more even dyeing was

obtained by means of the high temperature process.

DETERMINATION OF OPTIMUM DYEING CONDITIONS

Statement of conditions. In addition to evaluating the high temperature method of dyeing for fastness to light and washing, an effort was made to determine whether any saving could be made in the amount of time required for dyeing or in the amount of dyestuff used. To accomplish this purpose, additional dyeings were made in which the procedure for high temperature dyeing was followed except that the time of dyeing and the concentration of dyestuff were varied.

Variation of time of dyeing. In the previous dyeings at high temperature, the yarn was dyed at 250° F. for twenty minutes. In this test, the time of dyeing was reduced in increments of five minutes. For instance, separate dyeings were made in which the dyeing was continued at 250° F. for periods of twenty minutes, fifteen minutes, ten minutes, five minutes, and one dyeing in which the dyeing cycle was considered complete when the temperature of the dye bath reached 250° F. In addition, the dye bath was cooled to 180° F. in five minutes just prior to draining and rinsing. The cooling dye bath was utilized in anticipation of a more effective exhaustion of the dye bath. The exhaustion percentages for the dyeings performed under the time variables indicated are shown in Table 25.

Variation of dyestuff concentration. Another series of dyeings was made at high temperature under pressure in which dye concentrations of 2.0 per cent, 1.75 per cent, 1.50 per cent and 1.25 per cent were used. Again the dye bath was cooled to 180° F. after dyeing was complete and just prior to draining and rinsing to effect better dye bath exhaustion. The exhaustion percentages for these dyeings are shown in Table 26.

Fastness tests. The yarn dyed under the specified variables mentioned above was subjected to the same types of fastness tests as the yarn dyed at 250° F. for twenty minutes in a concentration of two per cent dyestuff. Knitted samples of the dyed yarn were exposed in the Atlas Fadeometer for twenty, thirty, forty and fifty hours. Knitted samples were also subjected to A.A.T.C.C. Test No. 2.

CHAPTER V

RESULTS AND DISCUSSION OF RESULTS

Breaking strength tests. The average breaking strength of the undyed yarn was determined to be 1.26 pounds. An analysis of the breaking strengths of the dyed yarns indicated that the average obtained for these samples was somewhat less. For yarn dyed at 212° F. with Pontamine Blue BB Super Concentrated, the average breaking strength was 1.10 pounds. For yarn dyed with the same dye at 250° F., the average breaking strength was also 1.10 pounds. Thus it is seen that these yarns suffered a decrease in breaking strength of 0.16 pounds, a loss of 12.70 per cent.

For yarns dyed at 212° F. with Pontamine Fast Rubine B Concentrated, the average breaking strength was found to be 1.07 pounds. For yarn dyed at 250° F. with this same dye, the average breaking strength was 1.08 pounds. These results indicate the yarns dyed with this dye suffered a decrease in breaking strength of approximately 0.19 pounds, a loss of 15.08 per cent.

The yarns dyed at 212° F. with Pontamine Fast Orange EGL exhibited an average breaking strength of 1.18 pounds, while the yarn dyed at 250° F. with this same dye had an average breaking strength of 1.21 pounds. This represented an average decrease in breaking strength of 0.06 pounds, or a loss of 4.76 per cent.

An analysis of the above results indicates that although the dyed yarns suffered a decrease in breaking strength ranging from 4.76

per cent to 15.08 per cent, the decrease for yarns dyed at 212° F. was virtually the same as for yarns dyed at 250° F.; and no portion of the decrease in tensile strength could be attributed to the use of high temperature procedures.

<u>Launderometer</u> <u>tests</u>. An examination of the samples subjected to A.A.T.C.C. Washfastness Test No. 2 revealed that the yarns dyed with Pontamine Blue BB Super Concentrated at 212° F. and at 250° F. showed appreciable fading, and thus must be rated as Class 1. The samples dyed with Pontamine Fast Rubine B Concentrated and Pontamine Fast Orange EGL showed very little fading when subjected to A.A.T.C. Washfastness Test No. 2 and should be rated as Class 3.

The degree of difference in shade noted in all of the dyed samples was retained in the faded samples. It is to be noted that whereas the manufacturer's rating of the latter two dyes was Class 4, the fact that a rating of Class 3 was obtained for the dyed samples can be attributed to the fact that a lower concentration of dyestuff was used in preparing these samples.

<u>Fadeometer</u> <u>tests</u>. Examination of the knitted samples subjected to exposure in the Fadeometer indicated that fading occurred after the following periods of time:

<u>Pontamine Blue BB Super Concentrated</u>

Samples dyed at 212° F.	1.25 hours
Samples dyed at 250° F.	1.25 hours

Rating: Class 1.

<u>Pontamine Fast Rubine B Concentrated</u>

Samples dyed at 212° F.	10.00 hours
Samples dyed at 250° F.	10.00 hours

Rating: Class 3.

Pontamine Fast Orange EGL

Samples dyed at 212° F.	20.00 hours
Samples dyed at 250° F.	20.00 hours

Rating: Class 4.

The degree of difference in shade noted in all of the dyed samples was retained in the faded samples, and the above results indicate that no improvement in fastness to light is imparted to cotton yarns dyed with direct dyes by the use of high temperature procedures.

Microscopic examinations. Fibers pulled from the dyed yarn samples and examined longitudinally under a microscope showed an apparently even penetration of dye into the fiber. The specimens observed showed uniform tinting throughout the length of the fiber.

Cross-section examinations indicated that penetration of the dyes was reasonably effective for all three dyes. However, those yarns dyed with Pontamine Fast Rubine B Concentrated exhibited more even penetration than the yarns prepared with the other two dyes.

The cross-sections were prepared by gathering a small bunch of yarns together and holding them tightly in a vise-like instrument. The projecting ends were frozen together in a rigid position with collodion. A tissue-thin slice was made across the diameter of the yarns and mounted in a glass slide. Microscopic examinations revealed that the fibers in the outer portion of the yarn received more dye than the inner portions since ring formations composed of heavily tinted fibers with more lightly tinted fibers in the center were distinctly visible in the yarns dyed with all three dyes at 212° F. and at 250° F.

Colorimetric examinations. Samples of the dye liquor in which Pontamine

Blue BB Super Concentrated was used at 212° F. in a solution of 2.0 grams of dyestuff in 16.0 liters of water indicated an exhaustion ranging from 0.0704 grams to 0.0824 grams. The average exhaustion obtained was 0.0750 grams or 31.52 per cent. Exhaustions ranging from 0.0280 grams to 0.0444 grams were obtained in using the same dye concentration at 250° F. The average exhaustion obtained when dyeing at 250° F. with this dye was 0.0383 grams or 16.64 per cent. The use of high temperature procedures with Pontamine Blue BB Super Concentrated resulted in a decrease in average exhaustion of 0.0367 grams or 14.88 per cent.

Dyeing at 212° F. with Pontamine Fast Rubine B Concentrated produced exhaustions ranging from 0.0720 grams to 0.0856 grams. The average exhaustion was 0.0798 grams or 32.20 per cent. Dyeing at 250° F. with this same dye produced exhaustions ranging from 0.0968 grams to 0.1248 grams. The average exhaustion obtained was 0.1114 grams or 44.38 per cent. The use of high temperature procedures with Pontamine Rubine B Concentrated resulted in an increase in average exhaustion of 0.0316 grams or 12.18 per cent.

The use of Pontamine Fast Orange EGL at 212° F. produced exhaustions ranging from 0.0960 grams to 0.1104 grams, the average being 0.1016 grams or 42.55 per cent. When dyeing with this same dye at 250° F., exhaustions ranging from 0.0544 grams to 0.0712 grams were obtained. The average exhaustion obtained at 250° F. was 0.0610 grams or 25.09 per cent. The use of normal high temperature procedures with Pontamine Fast Orange EGL resulted in a decrease in average exhaustion of 0.0406 grams or 17.46 per cent.

Optimum conditions of dyeing. Dyeing at 250° F. with Pontamine Fast Orange EGL with variations in time of dyeing from zero to twenty minutes in five-minute increments, together with cooling of the dye bath to 180° F. in five minutes, produced exhaustions ranging from 0.0944 grams to 0.1072 grams. This compares very favorably with the results obtained when dyeing with the same concentration at 212° F. in which the average exhaustion was 0.1016 grams.

Dyeing with Pontamine Fast Orange EGL at 250° F. with varying concentrations of dyestuff, together with cooling of the dye bath to 180° F. in five minutes, indicated a uniform decrease in exhaustion from 0.1008 grams for a 2.0 per cent concentration to 0.0656 grams for a 1.25 per cent concentration. However, the per cent exhaustion was approximately the same for these varying concentrations.

The samples produced by these variable dyeing conditions exhibited the same fastness qualities as the samples dyed under uniform conditions, which indicates that no reduction in fastness to light or washing is imparted by the dye deposited on the fibers through the introduction of the cooling dye bath. In addition the use of the cooling cycle increases the exhaustion of the dye bath and permits the use of a lower concentration of dyestuff to obtain approximately the same shade as that obtained by dyeing at 212° F. with a higher concentration.

CHAPTER VI

CONCLUSIONS

The following conclusions may be drawn from the results obtained in this study.

Breaking strength. Although the dyed yarns exhibited a loss in breaking strength of from 4.76 per cent to 15.08 per cent, the reduction in strength was noted in the yarns dyed at 212° F. as well as in those yarns dyed at 250° F. Therefore, it must be concluded that dyeing at elevated temperatures has no deleterious effect on the strength of the yarn, and the strength loss noted must have been imparted by some chemical action of the dyestuffs employed.

Washfastness. The results of the laundering tests indicate that the use of direct dyes at elevated temperatures imparts no additional resistance to fading by washing.

Lightfastness. The results of the light fading tests indicate that the use of direct dyes at elevated temperatures imparts no additional resistance to fading by light.

Dye penetration and exhaustion. The results obtained in this problem fail to indicate that additional penetration or exhaustion is obtained by dyeing with direct dyes at elevated temperatures. The increase in exhaustion in the case of one dyestuff and the reduction in exhaustion in the case of the other two dyestuffs can only be attributed to the chemical compositions of the dyes themselves.

Reduction in time of dyeing. The results obtained in this problem indicate that the overall time of dyeing at 212° F. can be reduced from approximately seventy-five minutes to approximately sixty minutes by the use of elevated temperatures and a cooling dye bath at the end of the dyeing cycle. This represents a twenty per cent reduction in time of dyeing.

Reduction in amount of dyestuff required. When dyeing at elevated temperatures with a cooling dye bath, visual examination of the samples indicated that a 1.75 per cent concentration produced the same shade as the 2.0 per cent dye concentration. This indicates a reduction of 12.5 per cent in the amount of dyestuff required to produce a given shade.

CHAPTER VII

RECOMMENDATIONS FOR FURTHER STUDY

The use of high temperature procedures offers a number of interesting possibilities for future investigations with regard to dyeing of cotton materials. Because of the results obtained in this problem with regard to dye exhaustion, it is recommended that an analysis of the composition of direct dyes be made in an effort to determine what effect the presence of various chemical components in the dyestuff has on the exhaustion of the dye bath at elevated temperatures. In addition, an effort should be made to utilize the results obtained in predicting the behavior of dyes of known composition.

Experimental work involving the use of high temperature procedures should be conducted in an effort to determine the effect of high temperature scouring on dyeing of cotton yarns. Another problem of interest would be the effect of high temperature dyeing on mercerized cotton materials.

The use of elevated temperatures could be applied to the differences in dyeability between mature and immature cotton fibers in an effort to determine whether or not the dyeability of immature fibers could be increased by the use of high temperature procedures.

BIBLIOGRAPHY

LITERATURE CITED

1. Cole, P. M., "The Barotor, A New Fabric Dyeing Machine", Papers Presented at the Technical Conference on Dyeing of "Orlon" Acrylic Fiber and "Dacron" Polyester Fiber. Wilmington: E. I. du Pont de Nemours and Company, Inc., 1952.

2. Zimmerman, Charles L., "The Application of Dyes to Textile Fibers at High Temperatures", American Dyestuff Reporter, 42 (September 14, 1953), pp. 627-634.

3. Drijvers, Ir. L., "Dyeing of Textile Fibers at High Temperatures (Above 100° C.)", Textielwezen, (August, 1952), pp. 18-37.

4. "A Closed High Temperature Dyeing Machine for Dyeing at Temperatures up to 135° C. (275° F.)", Textile Praxis, 3 (September, 1953), pp. 143-144.

5. "Belgian Dyeing Machine Uses Controlled Pressure", Textile World, 102 (December, 1952), p. 158.

6. Royer, G. L., and others, "Dyeing Studies at Elevated Temperatures Between 200° F. and 300° F.", Textile Research Journal, 18 (October, 1948), pp. 598-614.

7. Ibid., pp. 601-602.

8. Drijvers, op. cit., p. 27.

9. Lawton, Thomas W., and Paul C. Grant, Jr., "Cotton", Cotton Opening and Picking, Vol. 2B. Scranton, Pennsylvania: International Textbook Company, 1950, pp. 26-28.

10. Heuser, Emil, The Chemistry of Cellulose. New York: John Wiley and Sons, Inc., 1944, p. 3.

11. Vickerstaff, Thomas, The Physical Chemistry of Dyeing. New York: Interscience Publishers, Inc., 1950, p. 4.

12. Ibid., pp. 4-7.

13. Heuser, op. cit., p. 114.

14. Drijvers, op. cit., pp. 21, 23.

15. Heuser, op. cit., p. 114.

16. Drijvers, op. cit., pp. 21, 23.

17. Vickerstaff, op. cit., pp. 29-38.

18. 1953 Technical Manual and Yearbook of the American Association of Textile Chemists and Colourists. New York: Howes Publishing Company, Inc., 1953, pp. 90-92.

19. Ibid., pp. 108-111.

OTHER REFERENCES

Berkley, Earl E., "Cotton--A Versatile Textile Fiber", Textile Research Journal, 18 (1948), pp. 71-88.

Brauer, M., "Some Experiments with Sonic and Ultrasonic Waves in Dyeing", Melliand Textilberichte, 32 (1951), pp. 701-707.

Crank, J. J., "The Diffusion of Direct Dyes into Cellulose", Journal of the Society of Dyers and Colourists, 66 (1950), pp. 366-373.

Davidson, H. R., "Solution to the Artificial Light Problem", American Dyestuff Reporter, 41 (1952), pp. 1-10.

Eaton, J. C., C. H. Giles and Manfred Gordon, "Quantitative Relation Between Depth of Dyeing and Light Fastness", Journal of the Society of Dyers and Colourists, 68 (1952), pp. 394-396.

Jackson, J.H.E., and H. A. Turner, "The Desorption of A Direct Cotton Dye from Cellulose Fibers", Journal of the Society of Dyers and Colourists, 68 (1952), pp. 345-352.

Marone, A., "The Photoelectric Colorimeter in the Dyeing Technique and Its Application for the Control of the Whiteness of Bleached and Washed Products", Tinctoria, 43 (1951), pp. 316-318.

Millson, H. E., and L. H. Turl, "Microscopic Dyeing Phenomena; Studies with the Micro-dyeoscope", Textile Research Journal, 21 (1951), pp. 685-702.

Robinson, R. D., and C. L. Zimmerman, "Some Practical Aspects of High Temperature Dyeing", American Dyestuff Reporter, 39 (April, 1950), pp. 250-256.

Vickerstaff, Thomas, "Colorimetry Applied to Dyestuffs", Light and Lighting, 37 (March, 1944), p. 40.

Vickerstaff, Thomas, and D. Tough, "The Quantitative Measurement of Light Fastness", Journal of the Society of Dyers and Colourists, 65 (1949), pp. 606-612.

Ward, Kyle, Jr., "Crystallinity of Cellulose and Its Significance for the Fiber Properties", Textile Research Journal, 20 (1950), pp. 363-372.

Woods, H. J., "Structure of Fibers; Chain Molecules and Their Disposition; Crystalline and Amorphouse Parts of Fibers", Journal of the Textile Institute, 40 (1949), pp. 869-871.

APPENDIX

Table 1. Yarn Number or Count of Undyed Cotton Yarn.

Specimen Number	Weight of 120 Yards (Grains)	Yarn Number or Count
1.	66.74	14.98
2.	66.51	15.04
3.	67.30	14.86
4.	60.90	16.42
5.	63.46	15.76
6.	64.42	15.52
7.	65.54	15.26
8.	64.42	15.52
9.	62.06	16.11
10.	66.18	15.11
11.	60.48	16.53
12.	60.68	16.48
13.	62.30	16.05
14.	60.76	16.46
15.	65.56	15.25
16.	64.20	15.58
17.	67.28	14.86
18.	65.42	15.29
19.	65.48	15.27
20.	62.80	15.92
Total		312.27
Average		15.61

Table 2. Twists per Inch in Undyed Cotton Yarn

Specimen Number	Twists per Inch	Specimen Number	Twists per Inch
1.	21.70	11.	23.30
2.	24.35	12.	23.50
3.	24.25	13.	24.90
4.	23.75	14.	21.20
5.	24.10	15.	24.40
6.	24.10	16	24.90
7.	24.80	17.	21.90
8.	23.40	18.	24.90
9.	23.40	19.	23.80
10.	23.25	20.	23.20
Total			473.10
Average			23.66

Table 3. Single Strand Breaking Strengths of Undyed Yarn.

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.36	21.	1.18
2.	1.22	22.	1.34
3.	1.40	23.	1.28
4.	1.40	24.	1.32
5.	1.38	25.	1.40
6.	1.22	26.	1.20
7.	1.34	27.	1.34
8.	1.24	28.	1.14
9.	1.42	29.	1.10
10.	1.38	30.	1.12
11.	1.42	31.	1.22
12.	1.24	32.	1.22
13.	1.10	33.	1.34
14.	1.38	34.	1.12
15.	1.46	35.	0.92
16.	1.38	36.	1.20
17.	1.20	37.	1.15
18.	1.20	38.	1.26
19.	1.22	39.	1.31
20.	1.20	40.	1.14
Total			50.46
Average			1.26

Table 4. Light Transmission and Exhaustion Percentages for Cotton Yarn Dyed with Pontamine Blue BB Super Concentrated.

Sample Number	Per Cent Transmission Before Dyeing	Per Cent Transmission After Dyeing	Concentration of Dye (Grams/16000 c.c.) Before Dyeing	Concentration of Dye (Grams/16000 c.c.) After Dyeing	Exhaustion (Grams)	Exhaustion Per Cent
YARN DYED AT 212° F.						
1.	68.00	76.00	0.2360	0.1648	0.0712	30.17
2.	67.00	76.25	0.2464	0.1640	0.0824	33.44
3.	68.75	77.25	0.2304	0.1560	0.0744	32.29
4.	67.75	76.00	0.2416	0.1648	0.0768	31.79
5.	68.25	76.00	0.2352	0.1648	0.0704	29.93
Total					0.3752	157.62
Average					0.0750	31.52
YARN DYED AT 250° F.						
1.	68.75	73.00	0.2304	0.1888	0.0416	18.06
2.	69.00	73.75	0.2280	0.1856	0.0424	18.60
3.	68.75	72.50	0.2304	0.1860	0.0444	19.27
4.	68.25	72.00	0.2352	0.2000	0.0352	14.97
5.	69.00	72.00	0.2280	0.2000	0.0280	12.28
Total					0.1916	83.18
Average					0.0383	16.64

Table 5. Light Transmission and Exhaustion Percentages for Cotton Yarn Dyed with Pontamine Fast Rubine B Concentrated.

Sample Number	Per Cent Transmission Before Dyeing	Per Cent Transmission After Dyeing	Concentration of Dye (Grams/16000 c.c.) Before Dyeing	Concentration of Dye (Grams/16000 c.c.) After Dyeing	Exhaustion (Grams)	Exhaustion Per Cent
YARN DYED AT 212° F.						
1.	29.25	46.00	0.2496	0.1640	0.0856	34.29
2.	29.25	44.25	0.2496	0.1696	0.0800	32.05
3.	31.00	45.50	0.2368	0.1648	0.0720	30.41
4.	28.75	44.00	0.2520	0.1720	0.0800	31.75
5.	29.00	44.25	0.2512	0.1696	0.0816	32.48
Total					0.3992	160.98
Average					0.0798	32.20
YARN DYED AT 250° F.						
1.	28.50	49.25	0.2528	0.1488	0.1040	41.14
2.	31.00	51.75	0.2368	0.1400	0.0968	40.88
3.	28.00	53.75	0.2592	0.1344	0.1248	48.15
4.	28.50	52.50	0.2528	0.1376	0.1152	45.57
5.	29.00	53.25	0.2512	0.1352	0.1160	46.18
Total					0.5568	221.92
Average					0.1114	44.38

Table 6. Light Transmission and Exhaustion Percentages for Cotton Yarn Dyed with Pontamine Fast Orange EGL.

Sample Number	Per Cent Transmission Before Dyeing	Per Cent Transmission After Dyeing	Concentration of Dye (Grams/16000 c.c.) Before Dyeing	Concentration of Dye (Grams/16000 c.c.) After Dyeing	Exhaustion (Grams)	Exhaustion Per Cent
YARN DYED AT 212° F.						
1.	18.50	46.50	0.2360	0.1360	0.1000	42.37
2.	18.00	47.00	0.2384	0.1344	0.1040	43.62
3.	18.50	45.25	0.2360	0.1400	0.0960	40.68
4.	17.75	44.00	0.2400	0.1424	0.0976	40.67
5.	17.50	47.50	0.2432	0.1328	0.1104	45.40
Total					0.5080	212.74
Average					0.1016	42.55
YARN DYED AT 250° F.						
1.	17.75	30.50	0.2400	0.1824	0.0576	24.00
2.	17.50	29.00	0.2432	0.1880	0.0552	22.70
3.	17.50	28.75	0.2432	0.1888	0.0544	22.37
4.	17.25	33.00	0.2440	0.1728	0.0712	29.18
5.	17.25	32.00	0.2440	0.1776	0.0664	27.21
Total					0.3048	125.46
Average					0.0610	25.09

Table 7. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Blue BB, Super Concentrated. (Outer Portion of One-pound Package).

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.03	21.	0.83
2.	1.04	22.	0.97
3.	0.90	23.	0.82
4.	0.91	24.	0.95
5.	0.88	25.	0.88
6.	0.90	26.	1.31
7.	0.88	27.	1.00
8.	1.15	28.	0.96
9.	1.29	29.	1.20
10.	1.40	30.	1.18
11.	0.94	31.	1.18
12.	0.80	32.	0.90
13.	1.02	33.	1.18
14.	1.18	34.	1.46
15.	0.84	35.	0.88
16.	1.24	36.	1.10
17.	1.00	37.	1.18
18.	1.08	38.	1.36
19.	1.30	39.	1.08
20.	0.90	40.	1.24
Total			42.34
Average			1.06

Table 8. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Blue BB, Super Concentrated. (Middle Portion of One-pound Package).

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	0.83	21.	1.05
2.	1.38	22.	1.17
3.	1.54	23.	0.86
4.	1.41	24.	1.05
5.	1.00	25.	0.88
6.	1.25	26.	0.84
7.	1.33	27.	0.92
8.	1.17	28.	0.88
9.	1.00	29.	1.26
10.	1.42	30.	0.88
11.	1.34	31.	1.03
12.	1.13	32.	0.80
13.	1.20	33.	0.80
14.	1.20	34.	0.83
15.	1.13	35.	0.84
16.	1.20	36.	0.84
17.	1.38	37.	0.83
18.	0.89	38.	1.06
19.	0.79	39.	1.09
20.	1.30	40.	0.82
Total			42.62
Average			1.07

Table 9. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Blue BB, Super Concentrated. (Inner Portion of One-pound Package).

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.18	21.	1.06
2.	1.20	22.	1.37
3.	0.78	23.	0.84
4.	1.43	24.	1.17
5.	1.80	25.	0.90
6.	0.88	26.	0.89
7.	1.27	27.	1.09
8.	0.86	28.	1.18
9.	1.38	29.	1.24
10.	1.39	30.	1.12
11.	1.42	31.	1.55
12.	1.28	32.	0.95
13.	1.32	33.	0.91
14.	1.38	34.	0.99
15.	1.35	35.	1.18
16.	0.88	36.	1.44
17.	1.17	37.	1.13
18.	0.86	38.	1.02
19.	1.20	39.	1.35
20.	1.00	40.	1.35
Total			46.76
Average			1.17

Table 10. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F., Using Pontamine Blue BB, Super Concentrated. (Outer Portion of One-pound Package).

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.05	21.	1.15
2.	1.17	22.	1.24
3.	1.11	23.	1.19
4.	0.96	24.	1.38
5.	1.10	25.	0.99
6.	1.03	26.	1.12
7.	1.12	27.	0.73
8.	0.99	28.	1.12
9.	1.12	29.	0.96
10.	1.00	30.	0.95
11.	0.96	31.	0.95
12.	0.99	32.	0.92
13.	1.28	33.	1.12
14.	1.34	34.	1.13
15.	1.07	35.	1.14
16.	0.84	36.	1.47
17.	1.19	37.	1.26
18.	1.22	38.	1.08
19.	1.20	39.	1.20
20.	1.00	40.	1.24
Total			44.58
Average			1.11

Table 11. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F., Using Pontamine Blue BB, Super Concentrated. (Middle Portion of One-pound Package).

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.40	21.	1.22
2.	1.63	22.	1.40
3.	1.18	23.	0.80
4.	1.13	24.	0.77
5.	0.99	25.	1.09
6.	1.05	26.	1.34
7.	1.05	27.	0.95
8.	1.62	28.	1.36
9.	0.89	29.	1.35
10.	0.96	30.	1.29
11.	0.92	31.	1.26
12.	1.26	32.	0.70
13.	1.38	33.	1.02
14.	1.16	34.	1.16
15.	1.08	35.	1.22
16.	1.25	36.	1.17
17.	1.26	37.	0.90
18.	1.04	38.	1.09
19.	0.88	39.	1.10
20.	0.82	40.	1.00
Total			45.18
Average			1.13

Table 12. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Blue BB, Super Concentrated. (Inner Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.20	21.	1.29
2.	1.08	22.	1.16
3.	1.04	23.	1.00
4.	1.19	24.	0.84
5.	0.90	25.	1.05
6.	1.28	26.	1.18
7.	1.13	27.	1.00
8.	0.92	28.	1.39
9.	1.22	29.	1.34
10.	1.03	30.	1.06
11.	0.99	31.	0.96
12.	0.87	32.	1.42
13.	1.14	33.	1.18
14.	0.84	34.	1.02
15.	1.18	35.	1.04
16.	0.96	36.	0.84
17.	0.88	37.	0.82
18.	1.08	38.	1.02
19.	1.12	39.	0.82
20.	0.88	40.	1.25
Total			42.61
Average			1.07

Table 13. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Rubine B Concentrated. (Outer Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	0.99	21.	0.88
2.	0.82	22.	0.98
3.	1.09	23.	1.13
4.	1.00	24.	1.03
5.	1.24	25.	0.95
6.	1.00	26.	0.98
7.	0.78	27.	1.02
8.	0.99	28.	0.84
9.	1.06	29.	1.49
10.	1.09	30.	0.79
11.	1.18	31.	1.04
12.	1.34	32.	0.95
13.	1.25	33.	1.21
14.	0.92	34.	0.85
15.	1.36	35.	1.09
16.	1.51	36.	1.48
17.	0.88	37.	1.35
18.	1.05	38.	1.00
19.	1.44	39.	1.39
20.	0.92	40.	1.50
Total			43.86
Average			1.10

Table 14. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Rubine B Concentrated. (Middle Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	0.82	21.	1.18
2.	0.83	22.	1.32
3.	0.80	23.	0.88
4.	0.82	24.	0.86
5.	1.18	25.	0.80
6.	0.75	26.	1.33
7.	0.72	27.	1.21
8.	0.82	28.	0.82
9.	0.84	29.	1.25
10.	0.83	30.	1.29
11.	1.40	31.	0.98
12.	0.84	32.	0.88
13.	0.87	33.	1.15
14.	1.35	34.	1.04
15.	1.00	35.	0.84
16.	0.82	36.	1.34
17.	1.27	37.	1.12
18.	0.82	38.	0.84
19.	0.96	39.	1.29
20.	1.05	40.	0.89
Total			40.10
Average			1.00

Table 15. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Rubine B Concentrated. (Inner Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	0.98	21.	1.26
2.	0.98	22.	1.26
3.	0.82	23.	1.62
4.	0.83	24.	1.26
5.	0.72	25.	1.20
6.	0.79	26.	1.09
7.	0.92	27.	1.02
8.	1.01	28.	0.95
9.	1.08	29.	1.25
10.	1.36	30.	1.13
11.	1.22	31.	1.06
12.	1.22	32.	1.18
13.	1.33	33.	1.18
14.	1.25	34.	0.94
15.	1.18	35.	0.85
16.	1.20	36.	1.06
17.	0.91	37.	0.96
18.	1.13	38.	1.25
19.	1.09	39.	1.03
20.	1.16	40.	1.44
Total			44.07
Average			1.10

Table 16. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Rubine B Concentrated. (Outer Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.27	21.	1.04
2.	1.37	22.	1.07
3.	1.68	23.	0.88
4.	1.52	24.	0.86
5.	1.26	25.	0.78
6.	1.34	26.	1.20
7.	1.61	27.	1.07
8.	1.21	28.	0.92
9.	1.25	29.	1.02
10.	1.35	30.	0.80
11.	0.82	31.	0.88
12.	0.88	32.	1.35
13.	1.35	33.	0.78
14.	1.18	34.	1.04
15.	0.83	35.	1.23
16.	1.12	36.	1.22
17.	1.18	37.	1.09
18.	1.06	38.	1.00
19.	1.02	39.	1.18
20.	0.87	40.	0.84
Total			44.42
Average			1.11

Table 17. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Rubine B Concentrated. (Middle Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.18	21.	0.81
2.	0.96	22.	0.98
3.	0.84	23.	0.90
4.	1.14	24.	0.82
5.	0.81	25.	1.10
6.	0.83	26.	1.02
7.	0.83	27.	0.98
8.	0.85	28.	0.89
9.	0.82	29.	0.82
10.	0.84	30.	0.86
11.	0.84	31.	0.90
12.	0.90	32.	0.89
13.	0.84	33.	1.45
14.	0.90	34.	1.64
15.	0.93	35.	0.97
16.	0.78	36.	1.00
17.	0.77	37.	1.42
18.	0.89	38.	1.42
19.	1.19	39.	1.60
20.	0.90	40.	1.08
Total			39.59
Average			0.99

Table 18. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Rubine B Concentrated. (Inner Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.12	21.	1.78
2.	1.12	22.	1.40
3.	1.48	23.	1.28
4.	0.84	24.	1.19
5.	1.34	25.	1.16
6.	0.96	26.	1.18
7.	1.18	27.	1.18
8.	1.18	28.	0.83
9.	1.51	29.	0.84
10.	1.41	30.	0.81
11.	0.97	31.	0.94
12.	1.17	32.	0.97
13.	0.90	33.	1.06
14.	1.35	34.	0.95
15.	0.80	35.	0.91
16.	1.24	36.	1.19
17.	1.17	37.	1.05
18.	1.20	38.	0.90
19.	1.46	39.	0.97
20.	1.45	40.	0.82
Total			45.26
Average			1.13

Table 19. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Orange EGL. (Outer Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds	Specimen Number	Breaking Strength (Pounds)
1.	1.09	21.	1.53
2.	1.07	22.	1.13
3.	1.26	23.	1.30
4.	0.94	24.	1.14
5.	0.94	25.	1.34
6.	1.17	26.	0.94
7.	1.08	27.	1.11
8.	1.19	28.	1.40
9.	0.99	29.	0.98
10.	1.25	30.	1.11
11.	1.06	31.	1.64
12.	1.02	32.	1.62
13.	1.04	33.	1.26
14.	1.06	34.	1.02
15.	0.91	35.	1.21
16.	1.17	36.	1.24
17.	1.41	37.	1.13
18.	1.32	38.	1.28
19.	1.07	39.	0.92
20.	1.14	40.	1.18
Total			46.46
Average			1.16

Table 20. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Orange EGL. (Middle Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.32	21.	1.03
2.	1.24	22.	1.27
3.	1.17	23.	1.40
4.	1.12	24.	1.08
5.	1.33	25.	1.26
6.	1.33	26.	0.96
7.	1.41	27.	0.99
8.	1.02	28.	1.22
9.	1.19	29.	1.12
10.	1.22	30.	1.22
11.	1.28	31.	1.31
12.	1.08	32.	1.20
13.	1.18	33.	1.03
14.	1.05	34.	1.18
15.	1.03	35.	1.10
16.	1.08	36.	1.11
17.	1.38	37.	1.13
18.	1.20	38.	1.25
19.	1.19	39.	1.26
20.	1.18	40.	1.38
Total			47.50
Average			1.19

Table 21. Single Strand Breaking Strengths, Cotton Yarn Dyed at 212° F. Using Pontamine Fast Orange EGL. (Inner Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.08	21.	1.25
2.	1.31	22.	1.06
3.	1.44	23.	1.14
4.	1.00	24.	1.04
5.	1.04	25.	1.33
6.	0.96	26.	1.18
7.	1.00	27.	1.25
8.	1.25	28.	1.17
9.	0.94	29.	0.95
10.	0.94	30.	1.14
11.	1.00	31.	1.10
12.	0.98	32.	1.06
13.	1.13	33.	1.39
14.	1.31	34.	1.18
15.	1.30	35.	1.50
16.	1.30	36.	1.30
17.	1.15	37.	1.20
18.	1.10	38.	1.40
19.	1.16	39.	1.68
20.	1.16	40.	1.53
Total			47.40
Average			1.19

Table 22. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Orange EGL. (Outer Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.31	21.	1.26
2.	1.26	22.	1.39
3.	1.44	23.	1.20
4.	0.93	24.	1.21
5.	1.26	25.	1.35
6.	1.03	26.	1.12
7.	1.41	27.	1.12
8.	1.38	28.	1.20
9.	1.35	29.	1.19
10.	1.37	30.	1.32
11.	1.25	31.	1.19
12.	1.22	32.	1.13
13.	1.10	33.	1.13
14.	1.34	34.	1.03
15.	1.17	35.	1.29
16.	1.28	36.	1.03
17.	1.36	37.	1.02
18.	1.16	38.	1.17
19.	1.01	39.	1.12
20.	1.03	40.	1.41
Total			48.54
Average			1.21

Table 23. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Orange EGL. (Middle Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.02	21.	1.29
2.	1.25	22.	1.33
3.	1.17	23.	1.16
4.	1.26	24.	0.83
5.	1.38	25.	1.31
6.	1.18	26.	0.99
7.	1.40	27.	1.35
8.	1.34	28.	1.20
9.	1.24	29.	1.25
10.	1.33	30.	1.30
11.	1.61	31.	1.20
12.	1.38	32.	1.17
13.	1.07	33.	1.12
14.	1.51	34.	1.11
15.	1.23	35.	1.15
16.	1.14	36.	1.11
17.	1.26	37.	0.83
18.	1.28	38.	1.14
19.	1.00	39.	0.84
20.	0.87	40.	1.32
Total			47.92
Average			1.20

Table 24. Single Strand Breaking Strengths, Cotton Yarn Dyed at 250° F. Using Pontamine Fast Orange EGL. (Inner Portion of One-pound Package)

Specimen Number	Breaking Strength (Pounds)	Specimen Number	Breaking Strength (Pounds)
1.	1.35	21.	1.25
2.	1.47	22.	1.56
3.	1.50	23.	0.96
4.	1.27	24.	1.27
5.	1.13	25.	1.06
6.	1.17	26.	1.17
7.	0.82	27.	1.09
8.	1.35	28.	1.10
9.	1.30	29.	1.40
10.	0.80	30.	1.09
11.	0.84	31.	1.14
12.	1.09	32.	1.31
13.	1.22	33.	1.58
14.	1.20	34.	1.26
15.	1.26	35.	1.41
16.	1.17	36.	1.38
17.	0.99	37.	1.35
18.	1.30	38.	1.02
19.	1.05	39.	0.98
20.	1.56	40.	1.20
Total			48.42
Average			1.21

Table 25. Light Transmission and Exhaustion Percentages for Cotton Yarn Dyed with Pontamine Fast Orange EGL Under Variable Conditions at 250° F.

	Per Cent Transmission		Concentration of Dye (Grams/16000 c.c.)		Exhaustion (Grams)	Exhaustion Per Cent
	Before Dyeing	After Dyeing	Before Dyeing	After Dyeing		
YARN DYED WITH VARIATION IN TIME:						
Time of Dyeing (Minutes)						
20	18.00	46.00	0.2384	0.1376	0.1008	42.28
15	18.50	44.75	0.2360	0.1408	0.0952	40.34
10	19.25	46.00	0.2320	0.1376	0.0944	40.69
5	18.25	44.25	0.2368	0.1424	0.0944	39.86
0	17.50	44.75	0.2432	0.2360	0.1072	44.08
Total					0.4920	207.25
Average					0.0984	41.40
YARN DYED WITH VARIATION IN CONCENTRATION:						
Concentration (Per Cent)						
2.00	18.00	46.00	0.2384	0.1376	0.1008	42.28
1.75	23.50	51.75	0.2112	0.1248	0.0964	45.64
1.50	30.25	62.50	0.1832	0.1048	0.0784	42.79
1.25	39.25	71.75	0.1552	0.0896	0.0656	42.27